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(54) COMPOSITE HEAT-SEALANT PARTICLES FOR INFUSION WEB MATERIAL AND METHOD FOR MAKING SAME

(71) We, THE DEXTER CORPORATION, a corporation organised and existing under the laws of the State of Connecticut, United States of America, of One Elm Street, Windsor Locks, Connecticut, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates generally to heat-sealable, infusion web materials. More particularly, it is concerned with a new and improved fibrous web material containing composite heat-sealant particles and with new and improved methods of providing both the web material and the composite heat-sealant particles.

It has long been known that fibrous web materials are well suited for use as infusion packaging material in the manufacture of tea bags and the like. Over the years a number of attempts have been made to impart to such web materials a heat-sealing character which would permit the heat bonding of the infusion package along its edges while at the same time avoiding interference with or impairment of the web's infusion characteristics. In such use it is essential to provide an efficient and effective bond capable of withstanding immersion in hot or boiling water, often for considerable periods of time. Originally this was accomplished by spreading a granulated or powdered thermoplastic heat sealant on one surface of a preformed porous web material and partially fusing the granular material, causing it to adhere to the web without destroying its porosity. The particulate thermoplastic resin generally employed was a copolymer of vinyl chloride and vinyl acetate readily available in powdered form. Unfortunately, the resin was not available as a fine powder and caused not only an extremely rough surface appearance but also produced aesthetically objectionable trans-

lucent spots dispersed across the web. Additionally, upon initial heating of the copolymer so as to obtain better adhesion of the resin to the fibrous web structure, the resin contracted, causing undesirable agglomeration or "balling" of the resin. Further, the fused resin tended to reduce the rate of liquid infusion through the web and frequently caused problems of resin buildup on the heat-sealing jaws of the sealing apparatus. Although the copolymer was also capable of being applied to the preformed web by discontinuous roller coating or similar techniques, materials so treated never met with substantial commercial success.

Subsequently there was developed a highly porous, waterlaid web material wherein the heat-sealable vinyl resin was added in fiber form. The thermoplastic fibers were about 1/16" in length and longer and were mechanically intertwined with the cellulosic fibers during the papermaking process thereby providing a degree of controlled intermingling within the web which was unattainable with the powder used theretofore. The heat-sealable fibers could be interspersed throughout the web but preferably were located predominantly at one surface of the sheet material. A more detailed disclosure of sheet material produced in accordance with this technique is disclosed in United States Patent No. 2,414,833 issued to Fay H. Osborne on January 28, 1947.

Although the vinyl copolymer resins used in the web materials were certified under appropriate FDA regulations, expert tea tasters were able consistently to detect minute traces of materials leached from the resin by the hot or boiling water used to make the tea. Accordingly, this detectable and objectionable organoleptic character coupled with a constant search for improved seal performance prompted attempts to employ other thermoplastics as the heat sealing component of such web material.

In this connection certain improvements

[Price 25p]

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were achieved over sheets containing coatings of copolymer powder by replacing the copolymer with polyethylene and similar thermoplastic polyolefin powders free of the undesirable organoleptic character. Since polyethylene has a specific gravity less than one, it is generally applied to a preformed web in powder form or as a uniform thin layer or sheet which is subsequently ruptured by heat shrinkage. Unfortunately, webs treated with such polyethylene powder possess many of the same disadvantages exhibited by earlier powder-coated web materials, such as a lack of powder retention on only one side of the web. Also, it is difficult to adequately fuse the powdered polyethylene to the web without at the same time causing heat degradation in the web.

Both powder and ruptured film techniques have consistently failed to provide the advantageous anchoring of the polyethylene to the body of the sheet structure in a manner similar to that obtained in the wet papermaking process described in the aforementioned Osborne patent. Attempts to incorporate polyethylene powder at the wet end of a papermaking machine in a manner similar to the aforementioned Osborne method have encountered many problems, not the least of which is the floatation of the material due to its low specific gravity.

Accordingly, it is an object of the present invention to provide a new and improved, porous, heat-sealable, infusion web material which combines many of the advantageous features of the fiber and powder heat-sealant materials while obviating many of the disadvantages thereof and at the same time providing improved seal strength with lesser amounts of heat-sealant materials.

Another object of the present invention is to provide a heat-sealable infusion web material incorporating heat-sealant particles exhibiting the beneficial properties of powdered heat-sealable material yet possessing the ability to be incorporated into and retained within the web during the conventional web-forming process. Included in this object is the provision for a web material exhibiting a smooth heat-sealable surface substantially free of the objectionable roughness or translucent spots heretofore found objectionable in webs incorporating vinyl copolymer powders.

A further object of the present invention is to provide a new and improved heat-sealable web material having improved hot seal strength, increased resistance to delamination in hot or boiling water and improved organoleptic character, all with lesser amounts of heat-sealant particles and without causing deterioration of the dry seal properties.

Another object of the present invention is to provide new and improved heterogeneous, composite, heat-sealant particles of attenuated nongranular structure adapted for use in

fibrous infusion web materials, which particles are characterized by an ability to be anchored with the fibers of the web materials and provide a secure mechanical bond therewith, yet at the same time retain the advantageous features associated with granular heat-sealable particulate materials.

Still another object of the present invention is to provide a new and improved water-dispersible, composite, heat-sealant particle of frail, delicate, attenuated, irregular, nodular-containing, chain-like appearance particularly well suited for use as the heat-sealing component of water-laid infusion fibrous web materials. Included in this object is the provision for a composite particle comprised of a solid-state dispersion of thermoplastic granules randomly implanted within and throughout an anomalistic network-like array of elongated, delicate, fiberless yet fibrilliform strands of another, preferably incompatible, thermoplastic carrier material.

A still further object of the present invention is to provide a new and improved heterogeneous composite particle of physically unchanged, granular, heat-sealable material capable of being incorporated into a fibrous web material at the wet end of a papermaking machine to impart to the web heat-sealing characteristics and improved seal strength, said granular material being encapsulated within and held by a delicate mesh of thermoplastic, carrier strands.

Another object of the present invention is to provide a new and improved composite heat-sealant particle including a granular component exhibiting the ability to be successfully incorporated into a wet papermaking process, said particles being free of autoadhesion at ambient temperatures below those required for heat sealing and exhibiting little or no contraction or shrinkage upon heating thereby avoiding agglomeration while at the same time imparting a smooth surface to the web material within which the particles are incorporated.

Still another object of the present invention is to provide a new and improved papermaking process well suited to the incorporation of powdered, low density, thermoplastic granules into a fibrous web at the wet end of a papermaking machine, the process providing more uniform distribution and intermixing of the powder within the fibrous web and excellent retention thereof by felting or mechanical entanglement. Included in this object is the provision for a wet papermaking process adapted for the aqueous distribution of thermoplastic powder having a specific gravity less than 1.0 while overcoming the tendency of the powder to float.

Another object of the invention is to provide a new and improved process for the production of heterogeneous composite particles comprised of granules entrapped in a sinuous or fibrous

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- carrier, the method including optimum and controlled operating conditions and parameters best suited for producing particles useful in a wet papermaking process.
- Still another object of the present invention is to provide a new and improved process for the production of composite thermoplastic particles of the type described in a rapid and efficient manner and the subsequent incorporation thereof into a waterfaid fibrous web at the wet end of a papermaking machine despite the fact that one component of the composite particles retains its individual granular characteristics. Included in this object is the provision for a process capable of producing resinous heat-sealant particles which minimize or substantially reduce the buildup of resin on heat-sealing jaws when paper incorporating the particles is used in the manufacture of infusion packages such as tea bags or the like.
- The object of the process is to produce the product.
- A further object of the present invention is to accomplish these various objectives in a simple and economical manner, preferably while using materials certified under appropriate regulations. Included in this object is the provision for imparting improved taste and appearance characteristics as well as excellent hot seal strength to heat-sealable infusion web materials while utilizing approximately one-third less heat seal material than has been utilized hereinbefore.
- Other objects will be in part obvious and in part pointed out in more detail hereinafter.
- It is hoped that the invention may go some way towards achieving some or all of these objects.
- A better understanding of the objects, advantages, features, properties and relationships of the invention will be obtained from the following detailed description and accompanying drawings which set forth certain illustrative embodiments and are indicative of the various ways in which the principle of the invention is employed.
- In the drawings:
- Fig. 1 presents a flow chart outlining a preferred method for producing the new and improved composite heat-sealant particles and infusion web materials of the present invention;
- Fig. 2 is an enlargement (38 \times magnification) of the composite heat-sealant particles utilized in the present invention; and
- Fig. 3 is a further enlargement (60 \times magnification) of heat-sealant particles produced by the process of the present invention clearly showing the granular component thereof implanted within the carrier component.
- A preferred embodiment will now be described.
- Turning to Fig. 1 of the drawings wherein the process for producing the composite heat-sealant particles and infusion web materials of the present invention is schematically illustrated in flow chart form, it will be noted that the attenuated composite heat-sealant particles are produced from two different thermoplastic materials by a technique involving concurrent precipitation and elongation. The composite particles are formed as flocculent precipitates characterized by the presence of elongated nongranular strands capable of being anchored to conventional papermaking fibers during web formation to provide the desired heat-sealable infusion web. The composite particles are not sheet-forming fibers and are incapable of forming independently self-sustaining waterleaves from 100% of the particles. Accordingly, they must be combined with other papermaking fibers in order to achieve the desired self-supporting fibrous web structure.
- As indicated in the flow chart, the carrier resin component is dissolved in a suitable solvent for that component and the insoluble granular resin component is subsequently dispersed in the carrier resin solution. A precipitant for the carrier resin is subjected to suitable flow conditions to produce the desired attenuation, such as rapid agitation, stirring or other shear conditions or simply rapid flow through a tubular precipitating chamber. The resin dispersion is added to the precipitant under these conditions and as the carrier resin is precipitated from solution it assumes the elongated fibrilliform structure seen in Fig. 2, carrying with it the undissolved thermoplastic granular component anchored therein. The carrier resin component embeds and entraps the granular resin component, securing it within the matrix of the elongated fiber-like precipitate. It is a feature of the present invention that the granules are physically unchanged during the entire precipitation process but are firmly and securely anchored within the carrier resin. The actual appearance of the heat-sealant particles is perhaps best seen in Fig. 3 wherein the discrete granules of undissolved resin can be seen positioned at isolated randomly spaced intervals throughout the carrier resin matrix. It will be noted that the carrier resin is not in the form of uniformly sized strands. Rather it varies substantially in both size and shape and appears to possess a multitude of fibril-like strands extending randomly both within and without its main structure. Since the granular component is randomly dispersed, some of the carrier resin strands embed the granular thermoplastic resins while others are completely free of any granular particles whatsoever. This high random fibrillatory construction renders the heat-sealant particles extremely well suited to retention in the papermaking process despite the fact that more than about 90% of the particles are less han 1/64" in their largest dimension and are incapable

of forming a self-sustaining waterleaf in the absence of other papermaking fibers. The particles can be readily dispersed in water and added to the wet end of a papermaking machine in much the same manner as conventional papermaking fibers but are preferably added in the manner indicated in the Osborne United States Patent No. 2,414,833 so as to form two-phase, heat-sealable, infusion web material. The particles might be thought of as being filtered from their aqueous dispersing media by the initial deposit of papermaking fibers prior to full web formation or complete drainage of the web. They preferably are positioned adjacent one surface of the web and impart the desired heat-sealing characteristics thereto. Advantageously the heat-sealant particles produced in accordance with the present invention exhibit uniquely beneficial properties such as substantially no shrinkage upon heating in the conventional paper-drying system, little or no tendency to agglomerate or self-adhere and resistance to detrimental web penetration at heat-sealing temperatures. The granular resin component constitutes about one-quarter and more of the particle's total weight and preferably about one-half the weight thereof so that the characteristics of the granular thermoplastic materials clearly evidence themselves in the final web during the heat-sealing process.

The normally solid, synthetic thermoplastic resins used as starting materials are not only different in composition but also generally exhibit a degree of incompatibility. More particularly, the thermoplastic material utilized as the granular component of the composite particles must be insoluble in a solution of the carrier resin. Stated differently, the solvent must be capable of dissolving the carrier resin while leaving the granular resin relatively unaffected, i.e., undissolved. Additionally, the carrier resin component must be capable of ready precipitation from the solvent under the requisite precipitaton conditions so as to produce the desired particle attenuation.

The flow chart indicates exemplary granular and carrier components which have given excellent results. However, these materials are set forth for illustrative purposes only and should not be considered a limitation of the invention. The vinyl copolymer carrier resin, preferably utilized is a copolymer of vinyl acetate and vinyl chloride. Examples of such material are the copolymeric material sold by Union Carbide Corporation under the trade designation "VYHH" or the copolymeric resin sold by Borden Company under the trade designation "VC-113". Similar materials have been used heretofore in fiber form in accordance with the teaching of the aforementioned Osborne patent. The thermoplastic vinyl copolymer is readily soluble in acetone and can be easily precipitated out of solution by addition to a suitable precipitant such as

water or a water-acetone mixture. The specific polymers employed are those certified under appropriate FDA regulations and particularly are those well suited in view of their heat-sealing characteristics. In general, normally solid, wholly synthetic, fiber-forming thermoplastic resins are employed so long as pairs or groups thereof are chosen which permit the desired composite granular-fibrous structure of the resultant heat-sealant particles. Thus, other resins such as polyamides or various nylon resins, polyvinyl compositions and copolymers, polyolefin compositions including polyethylene, polypropylene, and copolymers thereof and polyesters may be employed depending upon the particular end use desired.

For ease of illustration the invention will be specifically described with respect to the vinyl copolymers previously certified under appropriate FDA regulations for use in tea bags or the like. Accordingly, the preferred granular resin component will be a polyolefin composition of the polyethylene type while the carrier resin will be a copolymer comprised of about 86% vinyl chloride and 14% vinyl acetate. It will be appreciated that since the carrier resin is dissolved during the process of the present invention, the particular form of the carrier resin by the mere addition to

erial is not critical. It will also be appreciated that the particular solvent and precipitant will vary with the specific resins employed. In the preferred embodiment the solvent utilized is acetone, not only because of its solubility characteristics but also because of its adaptability to the precipitation process which permits precipitation of the carrier resin by the mere addition to water or a water-acetone mixture.

The granular resin component remains unchanged during the particle-forming precipitation process. Therefore, its size, configuration and properties can be selected to suit the end use desired. Preferably it consists of a fine powder of a thermoplastic synthetic resin wherein a major portion of the granules have a particle size substantially less than about 75 microns with about 40% or more of the material capable of passing through a 325 mesh U.S. standard sieve screen. The particle size distribution will vary from about 10 microns to about 100 microns with the preferred material being within the lower zone of this range. One such material used with good success is the polyolefin powder sold under the trade mark "Super Dylan 7120 (J-1)" by Koppers Chemical Company of Pittsburgh, Pennsylvania, and believed to be a copolymer of polyethylene and butylene. That material exhibits a specific gravity of 0.960 g./cc. at 23°C. (ASTM D-1505-63T), a melt index of 12.0 (ASTM D-1238-62T) and a softening temperature of 260°F. (ASTM D-1525-58T). The shape of the material is a relatively uniform spherical to spheroidal con-

figuration and the material exhibits a nonlinear or random molecular chain orientation. More than 85% of the powder passes through a 100 mesh screen, more than 80% passes through a 200 mesh screen and more than 40% passes through a 325 mesh screen.

The ratio of granular component to carrier resin component may vary substantially although generally it is preferred that the amount of granular component constitute more

than 20% by weight of the resultant composite heat-sealant particle, particularly where the particle is to be used in the manufacture of heat-sealable infusion web materials suited for tea packaging and the like. In this connection reference is made to Table I which shows the effect of a varying ratio of granular component to carrier resin component on the hot seal properties of the resultant infusion web material.

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TABLE I
Infusion Web * Properties for Different Composite Particle Compositions

Test	Ratio of Polyethylene to Vinyl Copolymer			
	10:70	20:80	25:75	55:45
Steaming, sec.	65	210	600+	600+
Dry Delamination, g./in.	260	305	260	—

* Two-phase webs made in accordance with United States Patent No. 2,414,833.
All webs contained about 30% by weight of heat-sealant particles.

As can be seen in Table I, a marked increase in the hot seal properties of the infusion web material takes place as the granular component concentration increases from 20% to 25% and above. These improved properties are obtained without adversely affecting other properties of the web materials.

Certain upper limits also can be placed on the amount of granular resin component in the composite heat-sealant particle. For example, in the wet papermaking operation it is undesirable to have a heat-sealant particle exhibiting a specific gravity less than 1.0. Accordingly, when polyethylene powder having a density of 0.960 is used as the granular component of the particle, and the vinyl copolymer carrier resin has a density of about 1.36, the upper limit of the granular component is about 85%. Above that percentage level the composite heat-sealant particles would exhibit a specific gravity close to or less than 1.0 and would tend to float. Actually an upper limit of approximately 70%—75% is used in practice and is preferred since the physical integrity of the heat-sealant particles is very poor between that level and 85% and can be disrupted quite easily by even the low degree of agitation occurring during the papermaking operation. This disruption results in a breakout of essentially free polyethylene granules with attendant problems of floatation and foam stabilization within the papermaking machine. Additionally, during the drying operation, the high percentage of granular component results in poor adhesion to the con-

ventional papermaking fibers of the infusion web and produces infusion web material having a low dry seal strength.

At the same time, it should be appreciated that the economics of the precipitation technique favor a high level of granular component since that leads to higher sealant particle to solvent ratios in the initial dispersion and higher output rates. Additionally, higher ratios of granular component result in low levels of self-adhesion during the removal of the sealant particle precipitate and a desirably finer particle size for the resultant heat-sealant particles.

At the opposite extreme, low levels of granular component have advantages in the papermaking operation and result in a heat-sealant particle which has greater integrity during the papermaking process and is more readily dispersible with little or no difficulty encountered due to floating material. However, as evidenced by the data set forth in Table I, a deterioration in the hot seal strength of the heat-sealant particle occurs when the granular component level dips below about 25%. Additionally, below this level the resultant binder exhibits a tendency to self-adhere and possesses a coarser or larger fibrous structure substantially exceeding the desired particle size limits to be discussed hereinafter.

It is a feature of the present invention that the optimum (preferred) ratio of granular component to carrier resin component has been found to lie within a range of about 1:3 to 3:1 to provide a system which re-

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tains the advantageous features of both the heat-sealant particle-producing process and the wet papermaking process. It will, of course, be appreciated that the exact ratio of the components will vary depending upon the particular starting materials employed, the economics of the particular precipitation process, the papermaking system utilizing the heat-sealant particles and the requirements of the infusion web being manufactured. However, the conditions set forth hereinabove have been found to be best suited to the production of infusion-type web materials for tea bags and the like.

The composite heat-sealant particles of the present invention are produced by adding the thermoplastic dispersion to a precipitant for the carrier resin under appropriate conditions of agitation. The precipitation process subjects the dissolved synthetic polymeric material to high shear conditions coincident with precipitation. This procedure effects disruption and elongation of the precipitate and results in attenuated, heterogeneously composite, precipitate particles. The dispersed granular component, which is physically unchanged by the carrier solution, is securely embedded or implanted within the carrier resin during its attenuated precipitation without loss of its granular characteristic. Although the shear precipitation technique is similar to the process previously used in the cellulose acetate industry and in the production of "fibrils", the composite heat-sealant particles of the present invention do not possess certain essential fibril characteristics. In particular, they neither form a web structure with wet strength integrity nor are they truly fibrous in form. They are incapable of forming a self-sustaining waterleaf from 100% of the particles. More specifically, webs formed solely from these particles cannot be removed from a web-forming wire and, when air dried, have little or no integrity.

It will, of course, be appreciated that the degree of agitation occurring during the precipitation of the heat-sealant particles may vary since fibril formation is not required. However, the attenuating force must be sufficient to impart to the precipitating resin an elongated fibrilliform nature or character capable of utilization within a papermaking process and an ability to be anchored within the web by conventional papermaking fibers. Although it has not been firmly established it is believed that the lack of shrinkage of the composite particle upon heating is attributable to the unoriented character of the carrier resin as formed during the precipitation process.

The concentration of the carrier resin in its solution prior to precipitation may vary somewhat but is generally maintained below about 35% by weight. In fact, the usual operating range is about 10%—30% with the preferred range for the vinyl copolymer being about 17%—20% by weight in acetone.

It has been found in accordance with the present invention that certain conditions of temperature and concentration of precipitant solution are required for best results. In the embodiment used for illustrative purposes wherein the carrier resin is dissolved in an acetone solution, a precipitant comprising a mixture of acetone and water may be used so long as the acetone constitutes less than about 50% of the precipitating solution and the solution is maintained at a temperature below room temperature, i.e., below about 25°C. Where one or both of these interrelated conditions are varied the resultant composite particle is affected. For example, it has been found that when the acetone concentration of the precipitant solution is below about 25% poor economies result in the recovery system. Therefore, the preferred operating concentration for the precipitant liquid is from 25% to 50% acetone with a typical concentration being about 38%.

As mentioned, temperatures exceeding 25°C. are undesirable. For example, when a temperature of 30°C. is used the particles become adhesive in nature, tend to agglomerate and produce particles of unduly large sizes, particularly at low granular component concentration levels. Preferably the temperature of the precipitant should be in the range of about 0°C. to 5°C. These temperatures provide good dispersion of the precipitate and facilitate filtration of the resultant composite particles. Accordingly, the preferred precipitant solution is one containing about 38% acetone and maintained at a temperature of about 0°C. Under these conditions the composite heat-sealant particles remain dispersed and do not exhibit undesirable self-adhesion.

As mentioned hereinbefore, the size of the composite heat-sealant particles will vary depending on a number of factors. Generally the particle size should be suited to the papermaking process and the ability of papermaking machinery to handle dispersions of the particles in the manner illustrated in the Osborne patent. Large particles generally yield poor dispersions in the papermaking system and exhibit poor felting and low adhesion to the fibers in the web during the conventional paper-drying process. Additionally, the large coarse particles produce a relatively rough surface, tend to flatten out (coining effect) during processing, and can cause objectionable translucent spots in the seal area of the infusion web material. The fine particles, of course, give good dispersion during the papermaking process and have excellent felting and adhesion to the sheet during the drying process. The size of the particle will decrease with an increased shearing force during precipitation. This force can, of course, vary with the velocity of movement of the precipitant and the viscosity thereof. The time period during which the precipitate is highly plastic and

- deformable will effect the particle size of the resultant heat-sealant particles. By lengthening the precipitation time, such as by the use of a precipitant containing a high level of polymer solvent, one can give the shear forces a greater chance to disrupt the structure and produce a finer heat-sealant particle. Extremely fine particles, e.g., on the order of the polyethylene powder size, are undesirable as they tend to defeat the purpose of the invention.
- The particles are conveniently classified by the Bauer-McNett classification system. The separation is done on an aqueous dispersion of particles and is reported as the percentage by weight retained on different size screens. Generally, particles retained on a 14 mesh screen are undesirable for use in the manu-
- facture of heat-sealable infusion web materials for the reasons given hereinbefore; however, up to about 1% of such particles can be tolerated. Particles retained on a 35 mesh screen are deleterious but to a lesser extent and up to about 10%—15% can be tolerated. Accordingly, the conditions of precipitation should be controlled so that substantially none of the particles are retained by a 14 mesh screen and less than about 10% of the particles are retained on a 35 mesh screen. Of the remaining 90%, a conventional size distribution is preferred with a 150 mesh screen constituting a typical midpoint size. A pair of exemplary heat-sealant particle size distributions producing excellent results are set forth in Table II.

TABLE II
Composite Particle — Size Distribution*

Screen Mesh	Size of Screen Openings (mm.)	Per Cent Retained	
		A	B
14	1.17	0.2	0.3
35	0.42	1.3	10.7
150	0.10	35.8	56.2
Pan	—	62.7	35.8

* Bauer-McNett Classification.

- It will, of course, be understood that the particle size produced during the shear precipitation process can and often is reduced by the use of normal attrition equipment employed in the papermaking industry, such as the well known Jordan pulp refiner. In fact, it has been found that the high hydraulic shear encountered in such a machine is preferred over the metal-to-metal contact found in other standard attrition equipment such as a Hollander Beater. In this connection, Table III sets forth the effect of particle size on the properties of heat-sealable infusion paper produced from those materials.

TABLE III
Effect of Sealant Particle Size on Properties of Infusion Paper*

	Particle Size	
	Retained on 35-Mesh	Through 35-Mesh and Retained on 150-Mesh
Steaming, sec.	400	600+
Dry Delamination, g.	210	210
Seal Penetration, in. g./in. ²	195	2

* Sealant particles composed of a polyethylene/vinyl copolymer in the weight ratio of 55:45. Classification made on a Bauer-McNett Classifier. Two-phase sheets were produced using a nominal 10-pound basis weight web of which about 3 pounds were sealant particles.

- As shown in Table III, the particles retained on a 35 mesh screen did not exhibit hot seal properties as good as those exhibited by particles small enough to pass through the 35 mesh screen but large enough to be retained by a 150 mesh screen. The seal penetration data is particularly noteworthy. In that test high values indicate that the thermoplastic sealant resin penetrates the fibrous web structure to an excessive degree. Such penetration will cause a buildup of resin on the heat seal jaws of a sealing machine. Therefore, it is clear that a large percentage of particles retained on a 35 mesh screen would be undesirable. These effects are even more pronounced with material retained on a 14 mesh screen.
- Although the lower limit on the particle size may vary, it will be appreciated that the particles must be large enough to be retained by the fibrous base web during the manufacture of the infusion web material. It should be understood that the particle size set forth herein relates directly to the production of infusion web material for use in the coffee and tea product area and that other areas exist where the presence of larger particles would not be deleterious.

- For a better appreciation of the appearance of the heterogeneously composite particles reference is made to Figs. 2 and 3 which show typical particles of the present invention. Fig. 2 clearly evidences the attenuated fibrilliform character of the flocculent precipitate while Fig. 3 further shows the chain-like, nodular-containing strands characteristic of the composite particles. As will be appreciated the shapes of individual particles vary substantially and quite randomly, and they clearly evidence

an elongated nongranular character. After precipitation the composite heat-sealant particles are usually filtered and thoroughly washed prior to being redispersed in water for use at the wet end of a papermaking machine.

As mentioned hereinbefore the new and improved heat-sealable infusion web material of the present invention is produced in accordance with the papermaking technique described in the Osborne United States Patent No. 2,414,833. In that method a dilute dispersion of heat-sealable, vinyl copolymer fibers of 1/16" length or longer is added to a furnish of non-thermoplastic papermaking fibers as those fibers are being deposited on the paperforming wire. The vinyl copolymer fibers are preferably introduced into the head box of a papermaking machine for deposit on the paperforming wire at an intermediate point along the wire's path of fiber collection.

The arrangement of the papermaking machine used in the present invention is substantially the same as set forth in that patent and the consistency of the composite particle dispersion of the present invention is approximately the same as the consistency utilized heretofore. The dispersion is added to the papermaking fiber furnish in such a manner as to permit intermingling of the particles and fibers and deposition of both on the paperforming wire of the papermaking machine. As pointed out in the aforementioned Osborne patent, the non-thermoplastic papermaking fibers generally start to deposit on the paperforming wire at a point in advance of the point of introduction of the heat-sealant particles. In this way a first phase or initial fiber layer is formed on the paperforming wire of the machine so that one surface of the resultant

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- infusion web material is composed of completely non-thermoplastic papermaking fibers. In this way the first phase of the web material may act as a base for receiving and anchoring the composite particles. The fibers continue to deposit with the particles as the papermaking wire moves through the head box thereby assisting in the deposition and retention of the particles. Although some papermaking fibers may be present on the top surface of the resultant web, it is comprised predominantly of the composite heat-sealant particles. It will, of course, be appreciated that the concentration of composite particles decreases through the depth of the sheet. In a manner of speaking, the initial deposit of fibers acts as both a filter bed for the particles and as a base for receiving additional fibers. It should be kept in mind that even the initial deposit of fibers is extremely dilute and continues to take part in the dynamics of the system, readily interferting with subsequently deposited fibers. Although no clear line of demarcation separates the two phases of the web, the bottom surface thereof is essentially devoid of thermoplastic particles. This is particularly advantageous when the sheet material is to be used on heat-sealing packaging machines. Such machines usually have heated jaws operating under pressure and it is important that the thermoplastic material be kept out of contact with the hot jaw to avoid resin buildup thereon and rupture of the seals produced.
- It is an additional advantageous feature of the present invention that substantially less thermoplastic material is required to produce the same and even improved heat seal properties. In particular, it has been found that the characteristics previously evidenced by web materials containing a certain amount of thermoplastic fibers are now achieved by the use of only two-thirds as much composite heat sealant of the present invention. As a specific example, if it is desired to produce an infusion web material having about seven pounds per ream (480 sheets, 24" by 36") of non-thermoplastic fibers, only about two pounds per ream of composite heat-sealant particles need be employed to obtain sealing characteristics previously requiring three pounds per ream of vinyl copolymer fibers.
- In view of the inability of the composite heat-sealant particles to form a waterleaf in the absence of papermaking fibers, it is generally preferred that the two-phase construction be utilized. However, complete intermixing of the particles with the conventional papermaking fibers also may be employed so long as the particles are retained to a sufficient degree within the sheet structure and are not washed therefrom during the papermaking process. Where the composite heat-sealant particles and papermaking fibers are thoroughly intermixed, it is frequently desirable to bondably secure the thermoplastic composite particles into the sheet of paper with highly beaten cellulosic fibers generally referred to as "flock". Other conventional binders may be employed with success.
- The infusion web materials resulting from the papermaking process are of thin, porous, lightweight construction but of sufficient strength to withstand the handling operations encountered in the manufacture of tea bags and the like. Such web materials generally have a basis weight of about 9 to 16 pounds per ream and are easily handled by the heat-sealing packaging machines.
- Having described the invention, the following examples are given for purposes of illustration only so that the invention may be more fully understood, and are in no way intended to limit the scope of the invention unless otherwise specifically indicated. All amounts are on a weight basis.
- Example I**
- A carrier resin solution was prepared by dissolving in acetone about 45 parts by weight of a vinyl copolymer resin (14% vinyl acetate and 86% vinyl chloride) sold under the trade designation "VYHH" by Union Carbide Corporation. To this solution was added about 55 parts by weight of polyethylene powder [Super Dylan 7120 (J-1) polyethylene powder] having a specific gravity of 0.960. The resultant dispersion was mixed to effect suspension of the polyethylene powder within the carrier resin solution and the solution was maintained at a temperature of about 29°C.
- A precipitant solution was prepared from about 38% acetone and 62% water and the temperature of the solution was reduced to about 0°C.
- Composite heat-sealant particles having an average size of about 100 microns and a composition by weight of about 55% polyethylene (specific gravity less than 1.0) and 45% elongated (attenuated fibrilliform) vinyl copolymer were produced from the aforementioned solutions in a constant flow precipitation tube. The precipitant solution was passed through a central tubular member at a rate of about 5.5 g.p.m. while the resin dispersion was introduced to the flowing precipitant solution at a rate of 0.4 g.p.m. through a hole located in the side of the tube. The composite fibrous particles produced therefrom were filtered, washed with water and redispersed for use in the papermaking process.
- The dispersion of heat-sealant particles was passed through a Jordan pulp refiner and fed to the head box of a papermaking machine substantially as described in Osborne United States Patent No. 2,414,833 at a rate sufficient to result in a heat-sealant particle content of about 20% by weight of finished sheet or web. The resultant infusion web material had excellent appearance, improved hot wet

- seal strength and organoleptic character and was comparable in all other properties to conventional heat-sealable infusion web material. The web material thus produced was used to 5 make tea bags on standard forming and sealing machinery and the resultant tea bags were found to have excellent hot wet seal properties.

Example II

The procedure of Example I was repeated 10 except that the polyethylene powder was replaced by polypropylene powder. (It is possible to replace it by any polymer or copolymer of ethylene and/or polypropylene, subject to its specific gravity being less than 1.0.) The 15 resultant heat-sealant particles were used to make a two-phase, heat-sealant infusion web material having a total thermoplastic particle concentration of about 30% of the total web weight. The resultant web was found to 20 have improved properties comparable to the web material of Example I.

As will be apparent to persons skilled in the art, various modifications, adaptations and variations of the foregoing specific disclosure 25 can be made without departing from the teachings of the present invention.

WHAT WE CLAIM IS:—

1. A thermoplastic heat-sealant material for imparting heat seal characteristics to a water-laid fibrous web material, said heat-sealant material being water-dispersible and adapted for incorporation into the web material at the wet end of a papermaking machine and comprising composite heterogeneous particles comprised of at least 20% by weight of a thermoplastic resin granular component dispersed throughout and securely embedded within an attenuated fibrilliform carrier matrix of another, incompatible, thermoplastic resin, the composite heterogeneous particles being incapable of forming a self-supporting waterleaf and having a particle size distribution permitting passage of 90% or more of the particles through a 35 mesh screen in accordance 45 with the Bauer-McNett classification system.
2. The heat-sealant material of claim 1 wherein the granular component is a polyolefin powder comprising up to 75% by weight of the particles, said powder combining with the carrier matrix to impart a chain-like, nodular-containing configuration to the particles.
3. The heat-sealant material of claim 1 wherein the carrier matrix consists essentially 55 of a copolymer of vinyl chloride and vinyl acetate.
4. The heat-sealant material of claim 1 wherein the granular component constitutes from 25 % to 75% of the composite particles 60 and includes a polyolefin powder having a specific gravity less than 1.0, a melt index of about 12.0, a substantially uniform spheroidal configuration, and a size distribu-

tion permitting passage of more than 80% through a 200 mesh screen and more than 40% through a 325 mesh screen.

5. The heat-sealant material of claim 1 wherein the composite particles exhibit an average size of substantially 100 microns and are comprised of substantially 55% by weight of thermoplastic polyolefin particles selected from the group consisting of polymers and copolymers of polyethylene and polypropylene having a specific gravity less than 1.0 and substantially 45% by weight of an elongated vinyl copolymer of attenuated fibrilliform configuration linkably securing the polyolefin granules in chain-like fashion.

6. A water-dispersible heat-sealant material suited for incorporation into waterlaid fibrous web material at the wet end of a papermaking machine comprising composite heterogeneous particles comprised of a granular thermoplastic resin component dispersed throughout and securely embedded within an attenuated fibrilliform carrier matrix of another incompatible thermoplastic resin, the particles being incapable of forming a self-supporting waterleaf, said granular resin component having a specific gravity less than 1.0 and the carrier resin component a specific gravity greater than 1.0, the proportion of granular resin component to carrier matrix being such as to provide the composite particles with a specific gravity greater than 1.0.

7. The heat-sealant material of claim 6 wherein the granular resin component is a polyolefin and the carrier resin component is a vinyl resin.

8. A waterlaid heat-sealable infusion web material comprising papermaking fibers and composite water-dispersible thermoplastic heat-sealant particles intermixed with the fibers and anchored within the web material, the composite particles being incapable of forming a self-supporting waterleaf and having a particle size distribution permitting passage of 90% or more of the particles through a 35 mesh screen in accordance with the Bauer-McNett classification system, said particles being comprised of a discrete granular thermoplastic resin component dispersed throughout and embedded within an attenuated fibrilliform carrier thermoplastic resin matrix, the two resins being incompatible.

9. The web material of claim 8 wherein the heat-sealant particles are concentrated at one surface of the web material and decrease in concentration through the depth of the web, the surface opposite said one surface being composed substantially of papermaking fibers.

10. The web material of claim 8 wherein the granular component of the particles has a specific gravity of less than 1.0 and is anchored to the web during web formation.

11. The infusion web material of claim 8 wherein the granular component includes thermoplastic polyolefin powder and the heat-

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- sealant particles are located predominantly adjacent one surface of the web. 45
12. The infusion web material of claim 8 wherein the granular component of the composite particles constitutes from 25% to 75% of the particles and the carrier matrix is a thermoplastic resin consisting essentially of a copolymer of vinyl acetate and vinyl chloride. 50
13. A waterlaid infusion web material comprising papermaking fibers and composite water-dispersible heat-sealant particles intermixed with the fibers and anchored within the web material, the composite particles having a specific gravity greater than 1.0 and being incapable of forming a self-supporting waterleaf, said particles being comprised of a discrete granular thermoplastic resin component having a specific gravity of less than 1.0 heterogeneously dispersed throughout and embedded within an attenuated fibrilliform carrier thermoplastic resin matrix, the two resins being incompatible. 55
14. The web material of claim 13 wherein the granular resin component is a polyolefin and the carrier resin component is a vinyl resin. 60
15. Process for producing heat-sealable infusion web materials useful in the manufacture of tea bags and the like, comprising the steps of forming aqueous dispersions of papermaking fibers and heat-sealant material and forming a web material therefrom, the process further comprising forming the dispersion of heat-sealant material by dispersing an insoluble granular thermoplastic resin component thereof in a solution of a carrier thermoplastic resin component thereof, the two resins being incompatible, and precipitating said carrier resin component under shear conditions in the presence of the granular component to form heterogeneous composite particles of a size and character incapable of forming a self-support- 65
- ing waterleaf, the particles being comprised of an attenuated fibrilliform matrix of the carrier component having the granular component dispersed throughout and embedded within the matrix. 70
16. The process of claim 15 wherein the carrier component is precipitated by adding the dispersion to a precipitant for the carrier component, said precipitant being at a temperature below 25°C. 75
17. The process of claim 15 wherein the heterogeneous composite particles include polyolefin granules having a specific gravity less than 1.0 present in an amount insufficient to cause flotation of the composite particles in the aqueous dispersion. 55
18. The process of claim 15 wherein the granular component has a specific gravity less than 1.0 and is dispersed in a solution of the carrier component in an amount sufficient to constitute 25% to 75%, by weight of the composite particles. 60
19. The process of claim 15 wherein fibers from the fiber dispersion are deposited as an initial dilute fibrous base web prior to deposition thereon of the heat-sealant particles to provide a two-phase infusion web material. 65
20. Materials as claimed in any one of claims 1 to 14, substantially as herein described with reference to Figs. 2 and 3 of the accompanying drawings. 70
21. Process as claimed in any of claims 15—19, substantially as herein described with reference to the accompanying drawings. 75
22. Product of the process claimed in any of claims 15—19 or 21.

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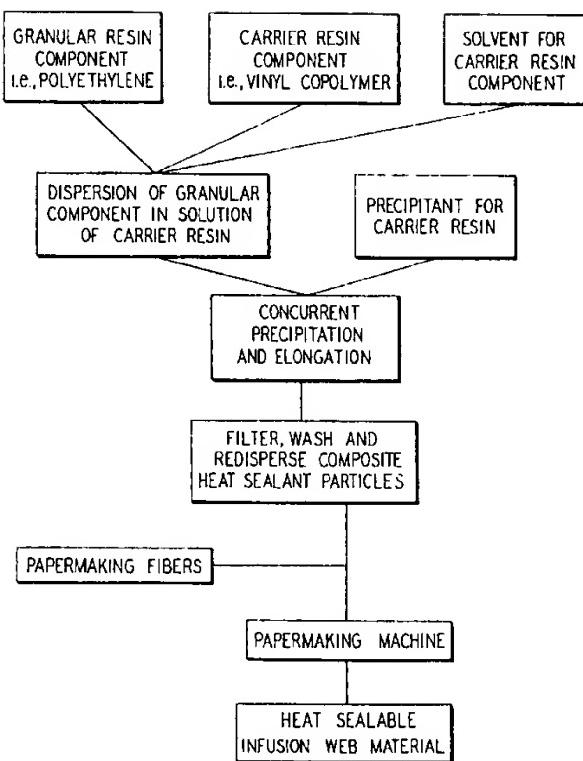
1297749 COMPLETE SPECIFICATION

2 SHEETS

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the Original on a reduced scale*

Sheet 1

FIG. 1



1297749 COMPLETE SPECIFICATION
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Sheet 2*

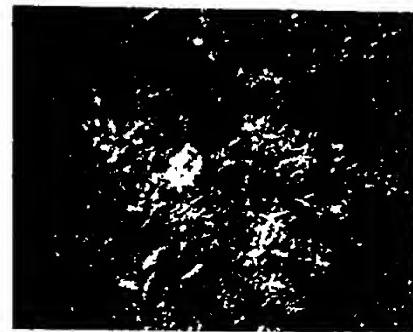


FIG. 2

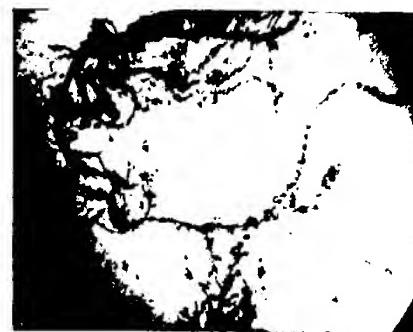


FIG. 3